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Effectiveness of Various Organometallics as Antiwear Additives in Mineral Oil

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EFFECTIVENESS OF VARIOUS ORGANOMETALLICS AS ANTIWEAR ADDITIVES IN MINERAL OIL

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SUMMARY

An investigation was conducted to examine the effectiveness of various types of organometallics in reducing the friction and wear of 1045 steel in sliding contact with 302 stainless steel. The organometallics were added in concentrations to 1 weight percent to degassed mineral oil. Sliding friction experiments were conducted in a pin or disk friction and wear apparatus. Wear tracks were subsequently examined with Auger emission spectroscopy for the detection of the elements present in the organometallics.

The results of this study indicate that there are organometallics which are equal in effectiveness to the commonly used zinc dialkyl dithiophosphate in the lubrication of surfaces. These substances include dimethyl cadmium, triphenyl lead thiomethoxide, and triphenyl tin chloride. All of the additives examined reduced friction from that observed with the degassed mineral oil, but only the aforenamed additivies reduced wear to the level observed with zinc dialkyl dithiophosphate. With dimethyl cadmium at concentrations of 0.5 weight percent and above, cadmium was detected in the wear track and, coincident with the first detection of cadmium, a marked decrease in sliding friction was observed.

INTRODUCTION

In recent years considerable attention has been focused on the antiwear additives incorporated in oils for industrial and automotive applications. The sulfur-phosphorus type of additive has largely replaced the earlier leaded oils; this is partly due to the better performance of the former, but it is also due to ecological and pollution consideration (ref. 1).

The friction, adhesion, and wear behavior of solids in contact can be altered markedly by surface films as thin as fractions of a monolayer (ref. 2). Thus, the study of the

chemistry of additive interaction with surfaces ought to be done with tools having sufficient sensitivity to detect such films. LEED (low energy electron diffraction) Auger emission spectroscopy analysis and ESCA (electron spectroscopy for chemical analysis) are ideally suited for such studies. These tools have been effectively used in recent tribological studies (refs. 3 to 6).

Alloys that are the most difficult to lubricate are the 300 series stainless steels. They, much like titanium alloys, exhibit a strong tendency to gall and seize. They are therefore ideally suitable as candidates for lubricant antiwear additive studies.

The objective of the present investigation was to examine in a controlled manner the influence on friction and wear of various antiwear additives with a 302 stainless steel surface sliding against a 1045 steel mating pin. Friction was measured during sliding, and the wear of the 1045 steel pin was measured after sliding. An Auger emission spectroscopy analysis (AES) of the wear track on the 302 stainless steel surface was made after each experiment. Various organometallics were added in 1 weight percent concentrations to a degassed pharmaceutical grade mineral oil. The lubrication experiments were conducted with a pin of the 1045 steel sliding on 302 stainless steel at a load of 1100 grams, a sliding velocity of 2.5 meters per second, and a temperature of 23° C. The sliding surface was immersed in the lubricant throughout each experiment, and an argon atmosphere was maintained above the lubricant.

MATERIALS

The rider specimens used in this study were 1045 steel with a 0.5-centimeter radius on one end. The disks were cut from 302 stainless steel shim stock.

All oil additives were reagent grade materials except the zinc dialkyl dithiophosphate, which was the commercially used material. The use of commercial grade zinc dialkyl dithiophosphate was by design to ensure a comparison of the other additives to practically used zinc dialkyl dithiophosphate.

The mineral oil was United States Pharmaceutical Grade. It was vacuum degassed by pumping on a flask of the oil with a mechanical pump.

APPARATUS

The apparatus used in this investigation is shown schematically in figure 1. It consisted essentially of a disk specimen 6.5 centimeters in diameter and a hemispherical rider specimen with a 0.5-centimeter radius on one end. The disk specimen was mounted directly to the end of a drive shaft of a electric motor. The disk sat in a metal pan 8.0 centimeters in diameter. The pan served as the lubricant reservoir.

The rider specimen was contained in an arm which was gimbal mounted to a support beam. The end of the arm opposite that containing the rider specimen was mounted by means of a flexible linkage to a strain gage assembly for friction force measurement. Friction force was continuously recorded during the experiment on a strip chart recorder.

The rider specimen was deadweight loaded against the disk surface. This was accomplished with a weight suspended from the arm containing the rider specimen.

The entire friction apparatus was enclosed in a clear plastic box in which a slightly positive argon pressure was maintained.

EXPERIMENTAL PROCEDURE

The 302 stainless disk specimen was polished with levigated alumina paste, rinsed with tap water followed by distilled water and finally ethyl alcohol, and then blown dry with dry nitrogen. The same procedure was followed for cleaning the 1045 steel rider specimen. The specimens were then mounted in the apparatus.

The lubricant was poured into the pan to a level just above the disk surface. Sliding was then begun with the full load on the rider. The experiment was run for 1 hour.

Upon completion of the experiment, the rider specimen was removed from the apparatus and the wear scar diameter was measured.

The disk was sectioned, rinsed with dichlorotetrafluoroethylene, and then placed in the vacuum system containing the Auger emission spectrometer. The disk surfaces and wear tracks were examined by Auger spectroscopy for those elements present. Auger spectroscopy samples the four to five outermost atomic layers giving an elemental analysis of the surficial layers.

RESULTS AND DISCUSSION

Sliding friction experiments were conducted with straight, additive-free mineral oil to obtain data for reference purposes. The data are presented in table I.

The coeffficient of friction measured for the metals lubricated with the additive-free mineral oil was very high, approximately 0.46. This is in the range of friction coefficients measured for the better friction materials (metals and alloys) in dry sliding.

The wear scar diameter was in excess of 2.6 millimeters. Both wear surfaces were extremely rough in surface texture. Metal transfer from rider to disk appeared to have occurred.

An AES analysis of the wear track revealed the presence of carbon, oxygen, and iron (fig. 2(a)). While the carbon is most likely due to the presence of a thin film of oil on

the disk, the oxygen and iron are due to the presence of iron oxide.

This iron oxide is believed to be wear debris from the 1045 steel rider because the principal oxide on 302 stainless steel dry is chrome oxide. Evidence for this is seen when the film on the disk surface is heated to 400° C for 10 minutes. The AES spectrum of figure 2(b) is of the same surface as shown in figure 2(a) after heating.

Examination of figure 2(b) indicates that the carbon peak has decreased in intensity, probably due to evaporation of the oil film in vacuum. Furthermore, chromium peaks appear in the spectrum on either side of the oxygen. These three peaks are generally seen on dry 302 stainless steel.

Zinc diaklyl dithiophosphate is one of the most commonly used extreme pressure additives. Sliding experiments were therefore conducted with 1-weight-percent zinc dialkyl dithiophosphate dissolved in the degassed mineral oil. The friction, wear, and AES results obtained with this lubricant are presented in table I. Table I shows that using zinc dialkyl dithiophosphate dissolved in degassed mineral oil reduces the friction and wear values below those obtained using additive-free mineral oil.

AES data obtained from the 302 stainless steel wear track after sliding and lubrication with zinc dialkyl dithiophosphate is presented in figure 3. The spectrum contains zinc, phosphorus, sulfur, carbon, chromium, and oxygen. All three of the elements zinc, phosphorus, and sulfur have friction and wear reducing ability. With all three present it is not certain which ones, if not all, are necessary to achieve the desired result of a reduction in friction and wear.

Attempts have been made to analyze films formed by using these additives to determine the mechanism of lubrication (refs. 7 and 8). Some have concluded that it is probably the sulfur (ref. 6). It is fair to say that the exact mechanism is, however, still not known.

Since zinc dialkyl dithiophosphate appears to elude analysis in its mechanism of lubrication two questions arise. First, are there other lubricant additives which are as effective in reducing friction and wear? Second, are there additives with a lessor number of active species that will give comparable lubricating effectiveness to that experienced with zinc dialkyl dithiophosphate and yet allow analysis?

Various materials were added in concentrations to 1 weight percent to mineral oil and then examined in friction and wear studies. Two of these additives, dimethyl cadmium and triphenyl lead thiomethoxide, gave very interesting results. The friction, wear, and AES results obtained with these additives are presented in table I.

Friction coefficients with dimethyl cadmium were slightly higher than those for zinc dialkyl dithiophosphate, but with triphenyl lead thiomethoxide lower. Dimethyl cadmium gave lower wear results than zinc dialkyl dithiophosphate.

An AES analysis of the 302 stainless steel disk wear surface after lubricated sliding with dimethyl cadmium is presented in figure 4. Three elements are detected - carbon from the oil and cadmium metal and oxygen from oxidized metal. Of these three ele-

ments cadmium is the only one that could account for the friction and wear results of table I. Carbon and oxygen were detected with the degassed mineral oil, but both friction and wear were markedly higher than was observed with dimethyl cadmium.

A comparison of the data in table I for zinc dialkyl dithiophosphate with dimethyl cadmium clearly indicates that a wealth of surface active additive elements are not necessary to achieve effective lubrication and wear reduction.

In table I both friction and wear were lower with triphenyl lead thiomethoxide than they were with zinc dialkyl dithiophosphate. The only surface active additive element detected aside from the ever present oxygen was lead after sliding with lubrication by triphenyl lead thiomethoxide as indicated in the AES spectrum of figure 5.

The interesting aspect of the data of table I and figure 5 is that with an additive containing both a metal and sulfur only metal deposits are in the wear area. The metal alone is as effective as or more effective than zinc dialkyl dithiophosphate where in sulfur interacts with the surface. This raises a word of caution against concluding that sulfur is the effective element in an additive where more than one element from that additive is found in the wear area.

Lead has been used as an antiwear additive in lubricants (e.g., lead naphthenate with active sulfur and/or chlorine) for some time. Cadmium has not. Further experimentation was therefore conducted with dimethyl cadmium.

One of the important practical considerations in the use of antiwear additives is the concentration of that additive necessary to provide a minimum in friction and wear. Various weight percentages of dimethyl cadmium were added to degassed mineral oil and lubrication experiments conducted with these formulations. The friction and wear results obtained are presented in figures 6 and 7.

In figure 6 the friction coefficient decreases markedly at concentrations in excess of 0.25 weight percent. At 0.75 and 1.0 weight percent the friction coefficients were comparable.

The rider wear results of figure 7 indicate a continuous decrease in wear to the 1045 steel rider with an increase in weight percent of dimethyl cadmium in the mineral oil. At 1.0 weight percent the wear scar diameter is less than was observed with 1.0-weight-percent zinc dialkyl dithiophosphate.

The AES analysis of the 302 stainless steel disk surface with the addition of various weight percent dimethyl cadmium yielded some interesting results. At concentrations up to 0.5-weight-percent dimethyl cadmium, only carbon and oxygen were detected in the wear track as indicated in the data of figure 8(a). The large carbon peak may be attributed to the presence of a oil film in the wear track. Heating the surface resulted in a diminution of the carbon peak height intensity.

The AES spectrum of figure 8(b) for 0.5-weight-percent cadmium indicates the presence of cadmium and oxygen on the surface. The same AES results were obtained with

greater concentrations of dimethyl cadmium; namely, cadmium and oxygen were present on the surface.

If the AES results are examined in light of the friction coefficients presented in figure 6, a relationship can be seen between the friction coefficient and the presence of cadmium on the surface. The sharp decrease in friction corresponds to the plating out onto the surface of cadmium from the dimethyl cadmium.

The relationship between cadmium deposition as detected by AES and wear is not as obvious. Examination of figure 7 does indicate a marked decrease in wear from 0.25 weight percent to 0.5 weight percent. Wear scar diameters were similar at 0.5 weight percent and 0.75 weight percent. One could conceivably see a break in the wear curve between 0.25- and 0.50-weight-percent dimethyl cadmium.

If the friction coefficient is examined as a function of time during sliding, insight into the additive surface activity can be gained. Frequently friction will decrease with time as additive-surface interactions take place. Both zinc dialkyl dithiophosphate and dimethyl cadmium were such effective boundary lubricants that this did not occur as indicated in the friction data of figure 9.

Data are presented in figure 9 for the straight degassed mineral oil, the oil containing 1-percent zinc dialkyl dithiophosphate, and the oil containing 1-percent dimethyl cadmium. With the degassed mineral oil the friction coefficient was very high (0.45) after 20 minutes of sliding. The presence of the additives in the oils brought about the very significant reduction in friction seen in figure 9. As significant, however, is the fact that with the additives the friction was low at the start of sliding and remained low during the entire period of sliding.

There are metals other than cadmium which may show promise as antiwear agents when incorporated in organometallics and added to oils. Tin has been used as a film to reduce friction, and bismuth appears to have desirable shear properties. Organometallic structures were obtained containing these elements and sliding friction experiments were conducted. Triphenyl bismuth and triphenyl methyl tin were added in concentrations to 1 weight percent to degassed mineral oil. Friction and wear results obtained with these lubricants are presented in table II. Results for the dimethyl cadmium are also presented in table II for reference purposes.

While the friction coefficient was reduced with the addition of triphenyl bismuth and triphenyl methyl tin from that observed with the additive free mineral oil, the wear was comparable to that obtained with the straight oil. Thus, these additives in concentrations of 1 weight percent did not offer any antiwear properties. Furthermore, with an AES analysis the bismuth could not be detected in the wear contact zone. Tin could, however, but this did not seem to assist in the wear reduction process.

Cobalt, even in elemental metal form, has been shown to have good friction and wear properties. Friction and wear experiments were conducted with an organometallic

containing cobalt, namely, cobalt carbonyl. The friction and wear results obtained are presented in table II.

Both friction and wear were reduced with cobalt carbonyl present in mineral oil from the results obtained with the additive-free oil. Cobalt was, however, not detected on the surface, only carbon as indicated in the Auger emission spectrum of figure 10. There are other metals which exist in a carbonyl structure, and some of these might have better antiwear qualities.

The tin containing organometallic triphenyl methyl tin in table II did not reduce wear. Many of the commercially used organometallic antiwear additives contain surface active nonmetallic elements. For example, zinc dialkyl dithiophosphate contains both sulfur and phosphorus. In addition to sulfur and phosphorus, many antiwear additives contain halogen atoms, particularly chlorine. Friction and wear studies were therefore conducted with triphenyl tin containing the halogens chlorine and bromine. Results of these experiments are presented in table III.

Both chlorine and bromine, when added to triphenyl tin, reduced wear. Triphenyl tin chloride reduced friction as well. An AES analysis of the wear surface indicates the presence of tin as well as the halogen. If the friction and wear results for triphenyl tin chloride (table III) are compared with the results obtained with zinc dialkyl dithiophosphate (table I) it can be seen that the results are comparable.

Carbon was detected in the AES analysis of all three organometallics examined in table III. The question arises as to the source of that carbon. Is it the mineral oil or the triphenyl group which gives rise to the carbon? Furthermore, does the organic portion of the organometallic play any role in the lubrication process? To answer this question sliding experiments were conducted with trimethyl tin chloride and triphenyl tin chloride in mineral oil. The friction, wear, and AES results obtained are presented in table IV.

In table IV the three elements carbon, tin, and chlorine were detected with an AES analysis with both organometallics. Differences in both friction and wear existed for the two additives. The triphenyl tin chloride produced lower friction and markedly less wear than was observed with the trimethyl tin chloride.

SUMMARY OF RESULTS

Based on the friction, wear, and AES results obtained in this investigation in the lubrication of 1045 steel sliding on 302 stainless steel with various organometallic additives in mineral oil, the following conclusions were reached:

1. There are additives which are just as effective in the lubrication of surfaces as is the commonly used commercial additive zinc dialkyl dithiophosphate. Such additives include dimethyl cadmium, triphenyl lead thiomethoxide, and triphenyl tin chloride.

2. All additives examined provided a reduction in friction when contrasted to the straight degassed mineral oil; however, only those additives indicated in the foregoing conclusion reduced wear appreciably.

3. AES analyses of dimethyl cadmium experiments indicated the presence of cadmium in the wear contact zone at concentrations of 0.5 weight percent and above. Coincident with the presence of cadmium a marked reduction in friction was observed.

4. With zinc dialkyl dithiophosphate, the active elements zinc, phosphorus, sulfur, and oxygen were detected in the wear contact zone while with triphenyl lead thiomethoxide, which gave comparable friction and wear behavior, only lead and oxygen were detected in the contact zone.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, August 30, 1977,
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TABLE I. - FRICTION, WEAR, AND ELEMENTS PRESENT IN WEAR TRACK OF 302 STAINLESS STEEL WITH VARIOUS LUBRICANTS

Lubricant	Coefficient of friction	Rider wear scar diameter, mm	Elements detected with Auger spectroscopy
Degassed mineral oil	0.46	2.613	Carbon, oxygen, iron, and chlorine
1 Percent zinc dialkyl dithiophosphate	.11	.605	Zinc, phosphorus, sulfur, carbon, chromium, and oxygen
1 Percent dimethyl cadmium[a]	.15	.410	Carbon, cadmium, and oxygen
1 Percent triphenyl lead thiomethoxide	.10	.580	Lead, oxygen

[a]In degassed mineral oil.

TABLE II. - FRICTION, WEAR, AND WEAR TRACK AUGER ELEMENTAL ANALYSIS OF 302 STAINLESS STEEL LUBRICATED WITH VARIOUS ORGANO-METALLICS IN MINERAL OIL

Lubricant	Coefficient of friction	Rider wear scar diameter, mm	Elements detected with Auger spectroscopy
1 Percent dimethyl cadmium[a]	0.15	0.410	Carbon, cadmium, and oxygen
1 Percent triphenyl bismuth[a]	.18	2.56	Carbon
1 Percent triphenyl methyl tin[a]	.16	2.61	Carbon and tin
1 Percent cobalt carbonyl[a]	.16	2.06	Carbon

[a]Additive in degassed mineral oil.

TABLE III. - FRICTION, WEAR, AND ELEMENTAL WEAR TRACK

ANALYSIS OF 302 STAINLESS STEEL LUBRICATED WITH

VARIOUS ADDITIVES IN OIL

1 Percent as additive in mineral oil	Coefficient of friction	Rider wear scar diameter, mm	Additive elements detected with Auger spectroscopy
Triphenyl methyl tin	0.16	2.61	Carbon and tin
Triphenyl tin chloride	.11	.58	Carbon, tin, and chlorine
Triphenyl tin bromine	.15	2.09	Carbon, tin, and bromine

TABLE IV. - FRICTION, WEAR, AND ELEMENTAL ANALYSIS

OF A WEAR TRACK ON 304 STAINLESS STEEL AFTER

SLIDING WITH TWO DIFFERENT LUBRICANTS

Lubricant	Coefficient of friction	Rider wear scar diameter, mm	Elements detected with Auger spectroscopy
1 Percent trimethyl tin chloride in mineral oil	0.18	2.34	Carbon, tin, and chlorine
1 Percent triphenyl tin chloride in mineral oil	.11	.58	Carbon, tin, and chlorine

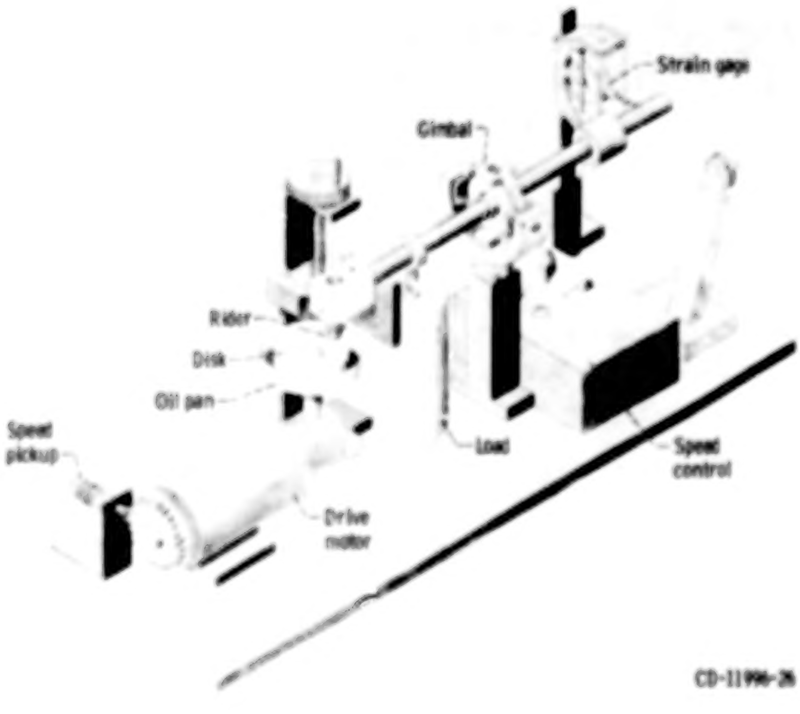
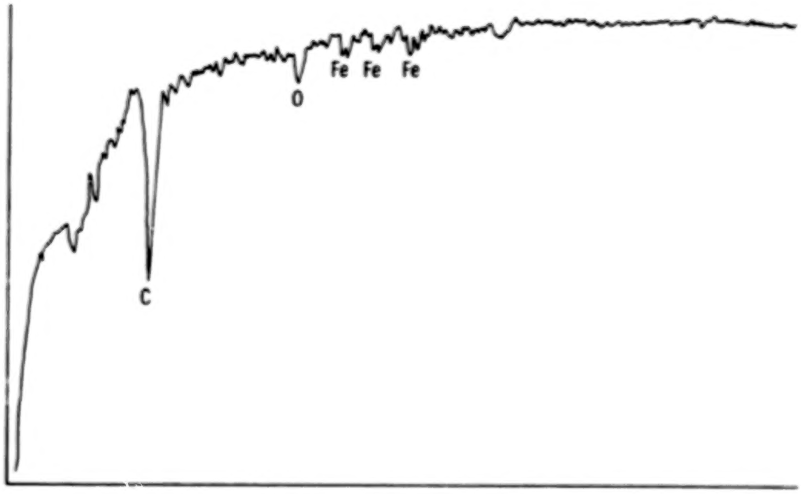
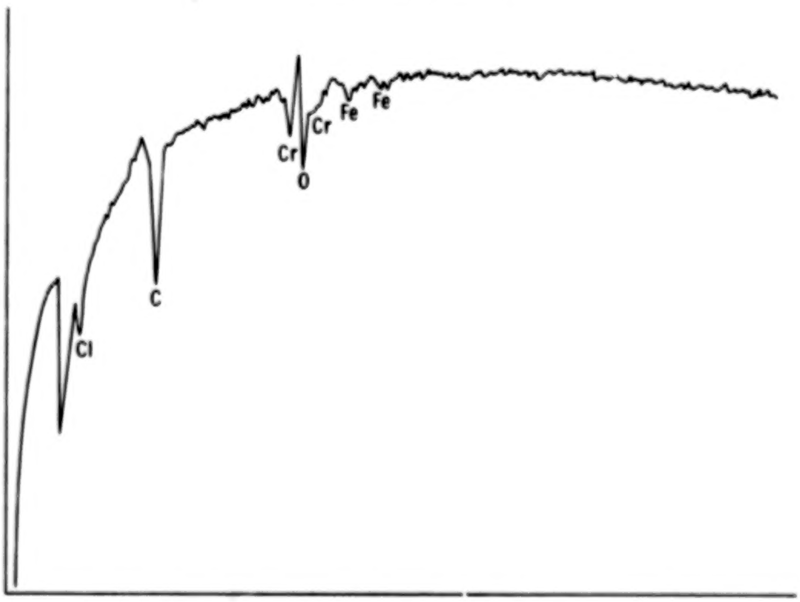


Figure 1. - Friction and wear apparatus.



(a) Initial surface condition.



(b) After sample had been heated to 400° C for 10 minutes.

Figure 2. - Auger emission spectroscopy trace of wear track on 302 stainless steel disk lubricated with degassed mineral oil.

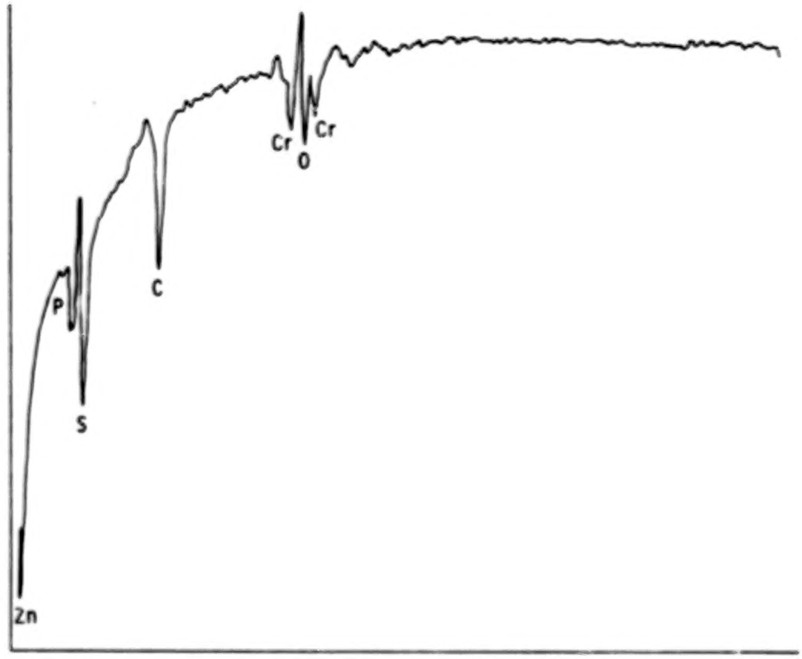


Figure 3. - Auger emission spectrum for 302 stainless steel wear surface after sliding with zinc dialkyl dithiophosphate additive in mineral oil.

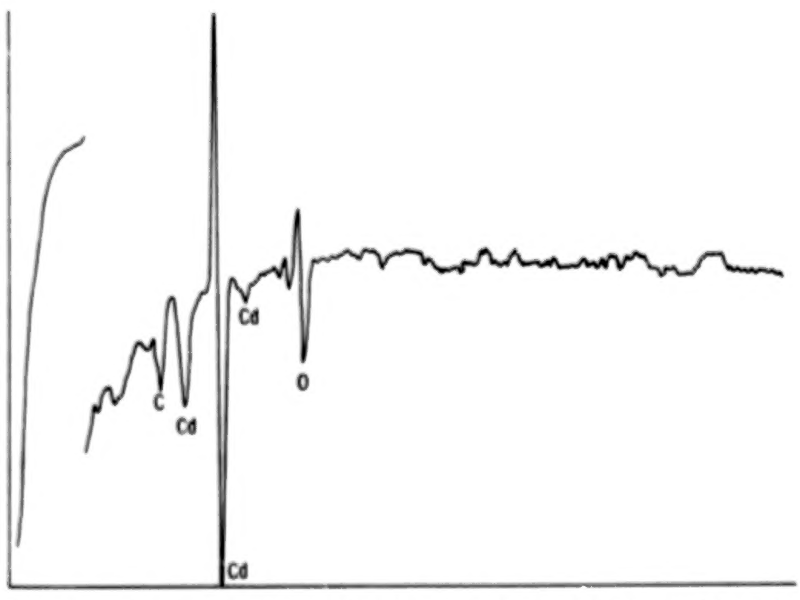


Figure 4. - Auger emission spectrum of 302 stainless steel lubricated with 1-percent dimethyl cadmium in mineral oil.

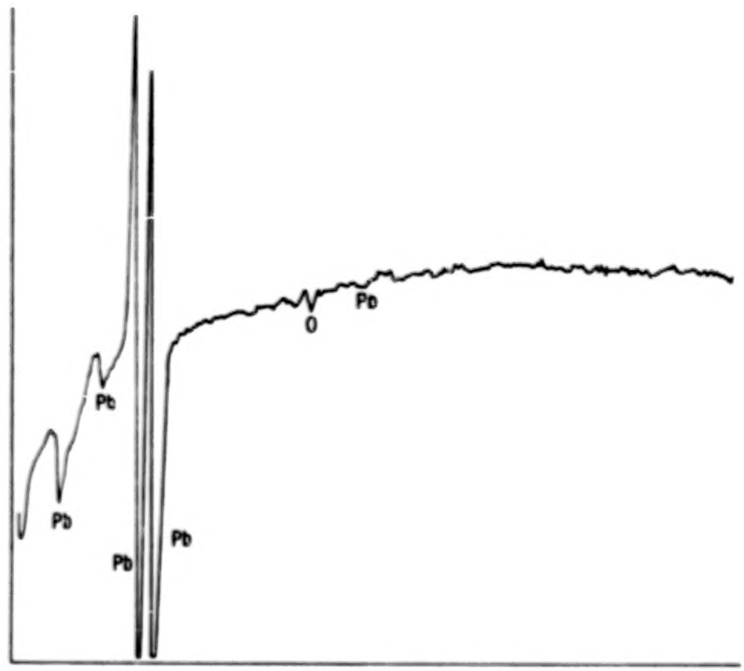


Figure 5. - Auger emission spectrum for 302 stainless steel lubricated with 1-percent triphenyl lead thiomethoxide in mineral oil.

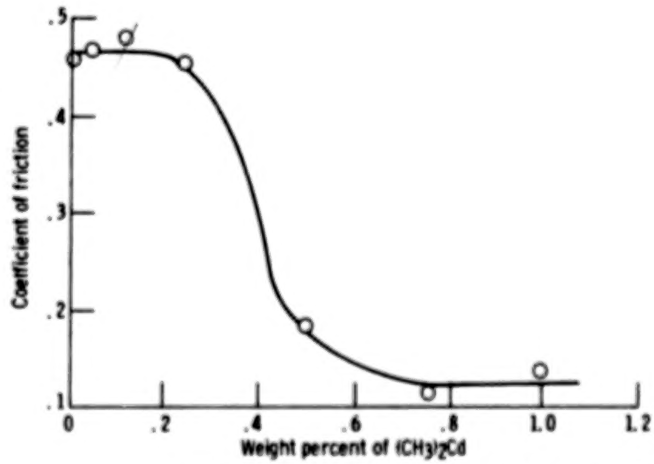


Figure 6. - Coefficient of friction for 302 stainless steel lubricated with various concentrations of dimethyl cadmium in mineral oil. Rider, 1045 steel; load, 1100 grams; sliding velocity, 150 meters per minute; temperature, 23° C.

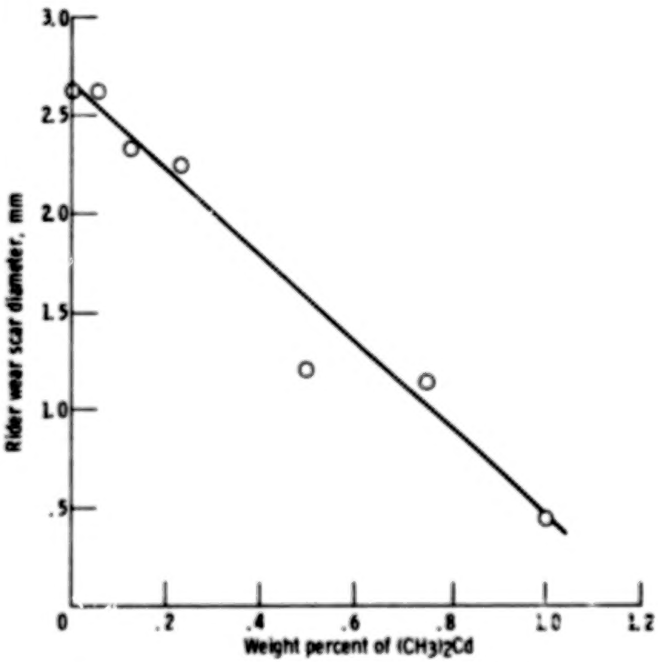


Figure 7. - Rider wear scar diameter for 1045 steel sliding on 302 stainless steel lubricated with various concentrations of dimethyl cadmium in mineral oil. Load, 1100 grams; sliding velocity, 150 meters per minute; temperature, 23° C.

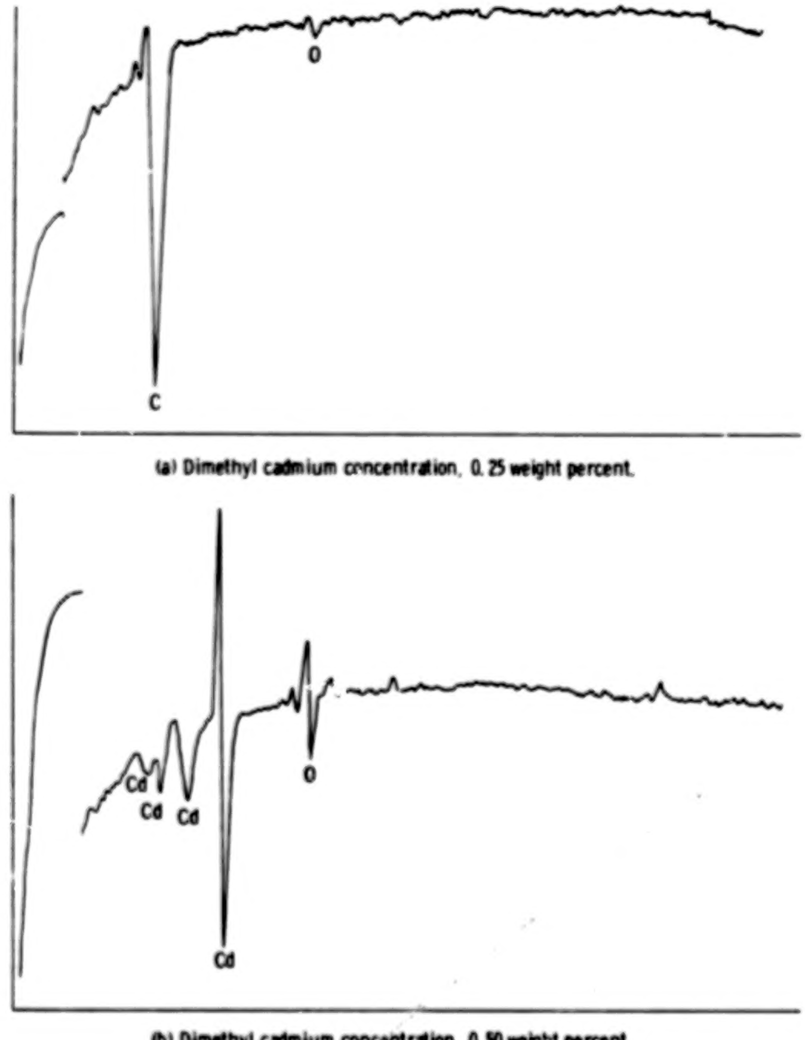


Figure 8. - Auger emission spectrum for 302 stainless steel lubricated with dimethyl cadmium in mineral oil.

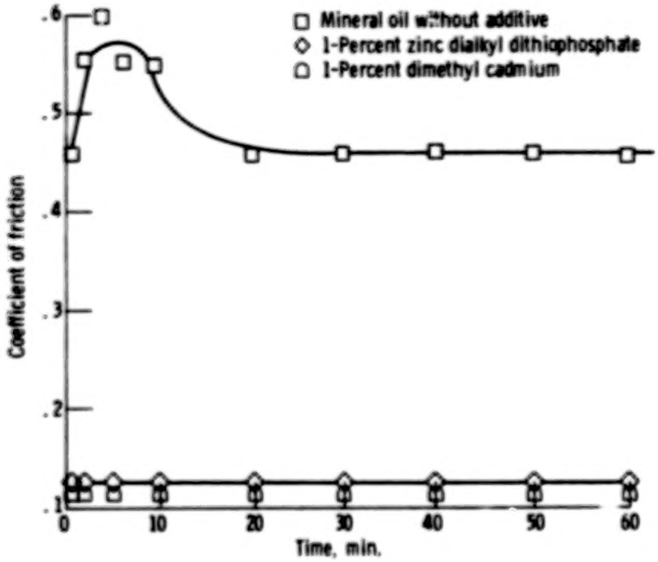


Figure 9. - Coefficient of friction as function of time for lubrication of 302 stainless steel with mineral oil and oil containing either 1-percent zinc dialkyl dithiophosphate or 1-percent dimethyl cadmium. Load, 1100 grams; sliding velocity, 150 meters per minute; temperature, 23° C.

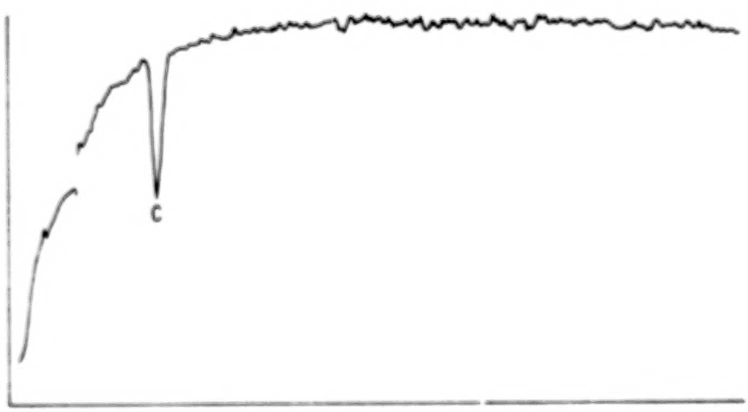


Figure 10. - Auger emission spectrum for 302 stainless steel lubricated with 1-percent cobalt carbonyl in mineral oil.

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16. Abstract

Sliding friction experiments were conducted with 1045 steel contacting 302 stainless steel and lubricated with various organometallics in mineral oil. Auger emission spectroscopy was used to determine the element present in the wear contact zone. The results indicate that there are organometallics which are as effective an antiwear additives as the commonly used zinc dialkyl dithiophosphate. These include dimethyl cadmium, triphenyl lead thiomethoxide, and triphenyl tin chloride. The additives were examined in concentrations to 1 weight percent. With dimethyl cadmium at concentrations of 0.5 weight percent and above, cadmium was detected in the contact zone. Coincident with the detection of cadmium, a marked decrease in the friction coefficient was observed. All additives examined reduced friction, but only the aforementioned reduced wear to a level comparable to that observed with zinc dialkyl dithiophosphate.

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